

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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CIRCULAR OF THE BUREAU OF STANDARDS, No. 203

RECOMMENDED SPECIFICATION FOR QUICKLIME AND HYDRATED LIME FOR USE IN THE MANU- FACTURE OF CALCIUM ARSENATE

[This is the tenth of a series of specifications for lime that is used in various chemical industries. To assist in the development of these specifications the bureau has called together an Interdepartmental Conference on Chemical Lime, composed of representatives of the Geological Survey and Bureau of Mines, of the Interior Department; the Bureau of Soils, Bureau of Chemistry, Forest Service and Fixed Nitrogen Laboratory, of the Department of Agriculture; and the Chemical Warfare Service, of the War Department. The present specification, based on a draft originally prepared by C. M. Smith, of the Board of Insecticides and Fungicides, Department of Agriculture, has been unanimously approved by the above conference and by the National Lime Association.]

ABSTRACT

Calcium arsenate is made by treating milk of lime with arsenic acid. For this purpose quicklime should be at least 92.5 per cent pure, hydrated lime 90 per cent, and should contain less than 1.5 per cent magnesia.

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I. GENERAL

1. USE OF LIME IN MAKING CALCIUM ARSENATE

The method most commonly used for the manufacture of calcium arsenate for insecticidal purposes consists in the addition of a solution of arsenic acid to a suspension of calcium hydroxide in water, under controlled conditions designed to give a product having certain

desired chemical and physical properties. The calcium hydroxide suspension is usually prepared immediately before use by the slaking of lump quicklime. Some manufacturers, however, purchase and use hydrated lime for this purpose.

2. DEFINITIONS

Quicklime is the product resulting from the calcination of limestone, and consists essentially of calcium oxide, or of calcium and magnesium oxides. It will slake when water is added to it, and this slaking is accompanied by an evolution of heat and an increase in volume. Hydrated lime is a dry powder which is made by treating quicklime with enough water to satisfy its chemical affinity under the conditions of manufacture. It consists essentially of calcium hydroxide, or of a mixture of calcium hydroxide and magnesium oxide or hydroxide.

3. PACKING

Quicklime is shipped in bulk in carload lots, in wooden barrels or metal drums holding 180 or 280 pounds each, or in waterproof bags holding 180 pounds. Hydrated lime is shipped in paper bags holding 40 or 50 pounds net each.

II. REQUIREMENTS

1. COMPOSITION

(a) AVAILABLE LIME.—Quicklime shall contain not less than 92.5 per cent available lime. Hydrated lime shall contain not less than 90 per cent available calcium hydroxide as computed by multiplying the per cent available lime by 1.3218.

(b) MAGNESIA.—Quicklime or hydrated lime shall contain not more than 1.5 per cent magnesium oxide.

The above percentages are based on the sample as taken at the point of shipment.

2. MARKING

Each carload of material or fraction thereof, shall be legibly marked with the names of the consignor and consignee, and with some means of identifying the particular contract on which the shipment is made. This information is in addition to that required by the Federal lime-barrel law.

III. SAMPLING AND TESTING

1. SAMPLING

When quicklime is shipped in bulk, the sample shall be so taken that it will represent an average of all parts of the shipment from top to bottom, and shall not contain a disproportionate share of

the top and bottom layers, which are most subject to changes. The sample shall consist of 1 shovelful for each 3 tons of material, but not less than 10 shovelfuls, taken from different parts of the shipment. The total sample taken shall weigh at least 100 pounds, shall be mixed thoroughly, and "quartered" to provide a 15-pound sample for the laboratory. In case a shipment consists of more than one car, a separate sample shall be taken from each car.

When quicklime is shipped in barrels, at least 3 per cent of the number of barrels shall be sampled. They shall be taken from various parts of the shipment, dumped, mixed, and sampled as specified in the above paragraph.

In the case of hydrated lime, the sample shall be a fair average of the shipment. Three per cent of the packages shall be sampled. The sample shall be taken from the surface to the center of the package. The material so obtained shall be thoroughly mixed and quartered to provide a 2-pound sample for the laboratory.

When sampling quicklime or hydrated lime, it is essential that the operation be conducted as expeditiously as possible, in order to avoid undue exposure of the material to the air. The sample to be sent to the laboratory shall immediately be placed in an air-tight container in which the unused portion shall be stored until the shipment has been finally accepted or rejected by the purchaser.

2. TESTING

(a) AVAILABLE LIME.—To 1.4 g of the carefully prepared and finely ground (passing a No. 100 sieve) lime in a 400 cc beaker add 200 cc of hot water, cover, heat carefully, and then boil for three minutes.

Cool, wash down cover, add two drops of phenolphthalein and titrate with normal hydrochloric acid, adding the acid dropwise as rapidly as possible, stirring vigorously to avoid local excess of acid. When white spots appear, retard the rate of addition of acid somewhat, but continue until the pink color fades out throughout the solution for a second or two. Note the reading and ignore the return of color.

Repeat the procedure of paragraph 1 above, using (instead of a beaker) a liter graduated flask carrying a one-hole stopper fitted with a short glass tube drawn out to a point. Cool, add slowly 5 cc less acid than before, stirring vigorously. Call the number of cc used "A." Grind up any small lumps with a glass rod slightly flattened at one end, dilute to the mark with freshly boiled distilled water, stopper, mix thoroughly for four or five minutes, and let settle for half an hour.

Pipette off a 200 cc portion, add phenolphthalein and titrate slowly with half-normal hydrochloric acid until colorless for one

minute. Call this additional number of cc "B." Then, percentage of available lime = $2A + 5B$.

(b) MAGNESIA.—The magnesia determination is made after removing silica, iron oxide, alumina, and lime. Ignite 1 g, or if the magnesium content is high 0.5 g, of the sample, in a platinum crucible over a blast for 15 minutes. Cool, moisten the residue, and dissolve it in 1:1 hydrochloric acid. Evaporate the solution to dryness on the steam bath, take up in 5 cc of hydrochloric acid, add 50 cc of water, warm until soluble salts are dissolved and filter off the silica. To the cool filtrate add 5 cc of hydrochloric acid, 1 g of ammonium persulphate, 5 drops of rosolic acid indicator and then ammonia until a distinct pink color appears. Heat the solution to boiling, boil for two minutes, filter and wash the precipitate with a hot 2 per cent solution of ammonium chloride. Acidify the filtrate slightly, heat to boiling, add ammonium oxalate slowly to excess, then ammonia slowly until in slight excess.

After the precipitate has settled for one to two hours filter and wash with an ammoniacal ammonium oxalate solution. Reserve the filtrate. Dissolve the precipitate in hot dilute hydrochloric acid and reprecipitate. Again filter the solution and wash the precipitate with a moderate amount of ammoniacal ammonium oxalate solution. Discard the precipitate, combine the filtrate with the first one, slightly acidify with hydrochloric acid, and add an excess of microcosmic salt. Then slowly add ammonia with stirring until a crystalline precipitate forms or the solution is alkaline. Finally add one-fifth of the volume of ammonia water in excess. After the precipitate has fully formed, preferably by standing overnight, filter and wash with dilute ammonia water (5 per cent by volume). Dissolve the precipitate in a slight excess of hydrochloric acid, add one or two drops of phosphate solution, and reprecipitate by adding ammonia while stirring, finally adding 2 or 3 cc of ammonia in excess. Let stand for 4 hours or overnight, filter, wash with 5 per cent ammonia, and ignite to constant weight. The weight of magnesium pyrophosphate $\times 0.3621$ = the weight of magnesium oxide.

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